WSC – CAM – V A

Quality Assurance and Quality Control Requirements for ***SW-846 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC)*** for the Massachusetts Contingency Plan (MCP)

Document Status: Final

Donald Muldoon
Department of Environmental Protection
One Winter Street
Boston, MA 02108
donald.muldoon@state.ma.us

Document Chronology

Revision	Status	Date
0	Public Comment Draft	5 October 01
1	Revised Public Comment Draft	17 December 01
2	Revised Interim Final	12 July 02
3	Final	1 May 03
4	Final	20 August 2004

V. Gas Chromatographic (GC) Methods

A. Quality Assurance/Quality Control (QA/QC) Requirements and Performance Standards for SW-846 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography

A.1 TABLE OF CONTENTS

1.0 QA/QC Requirements for SW-846 Method 8082 3
1.1 Method Overview 3
1.2 Summary of Method 6
1.3 Interferences 7
1.4 QA/QC Requirements for SW-846 Method 8082 8
1.5 MCP Analyte List for SW-846 Method 8082 16
1.6 Additional Analyte Reporting Requirements for SW-846 Method 8082 17
2.0 Data Usability Assessment for SW-846 Method 8082 18
3.0 Reporting Requirements for SW-846 Method 8082 19
3.1 General Reporting Requirements for SW-846 Method 8082 19
3.2 Specific Reporting Requirements for SW-846 Method 8082 19

List of Tables and Appendices

Number	Title	Page
Table V A-1	Performance Requirements for GC/MS Analyses by SW-846 Method 8082	11-15
Table V A -2	Analyte List for SW-846 Method 8082	18
Table V A-3	Routine Reporting Requirements for SW-846 Method 8082	19
Appendix V A-1	Sample Collection, Preservation, And Handling Procedures for SW-846 Method 8082 (PCB) Analysis	21
Appendix V A-2	GC/ECD Chromatograms of Aroclor 1262 and 1268, including Exhibits V A-1 and V A-2	22-23

1.0 QA/QC REQUIREMENTS FOR SW-846 METHOD 8082

1.1 Method Overview

SW-846 Method 8082 is used to determine the concentrations of polychlorinated biphenyls (PCBs), as Aroclors or as individual PCB congeners, in extracts from solid and aqueous matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target analytes may be determined by either a single- or dual-column chromatographic system. The method also may be applied to other matrices such as oils and wipe samples, if appropriate sample extraction procedures are employed.

1.1.1 Reporting Limits for SW-846 Method 8082

The reporting limit (RL) for SW-846 Method 8082 for individual Aroclors or congeners is instrument/detector dependent and also dependent on the choice of the sample preparation/introduction method and/or percent (%) solids of the sample. Using standard electron capture detection (ECD), the estimated Reporting Limit (RL) of Method 8082 for determining individual Aroclors is approximately 50 - 70 µg/kg (wet weight) for soil/sediment samples and 0.55 – 0.90 µg/L for aqueous samples. Somewhat higher RLs may be expected using electrolytic conductivity detectors (ELCDs). Reporting limits for SW-846 Method 8082 will be proportionately higher for sample extracts and samples that require dilution, or when a reduced sample size is used to avoid detector saturation.

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for PCBs analyzed in support of MCP decision-making are presented in Appendix V A–1 of this document and Appendix VII-A, WSC-CAM–VII A, “Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)".

1.1.2 Additional Requirements

Each laboratory that uses SW-846 Method 8082 is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and the analysis of laboratory control spikes (LCSs) and LCS duplicates to assess analytical accuracy and precision. Matrix spikes (MS), matrix spike duplicates (MSD) or Matrix duplicates may also be used to evaluate precision when such samples are analyzed either at discretion of the laboratory or at the request of the data-user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must

meet or exceed the performance standards as presented in Section 1.4 and Table V A-1 of this method. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 method 8000B (Section 8.4) and SW-846 method 8082 (Section 8.3). The data associated with the Initial Demonstration of Proficiency should be kept on file at the laboratory and made available to potential data-users on request. The data associated with the Initial Demonstration of Proficiency for SW-846 Method 8082 must include the following:

QC Element	Performance Criteria
Initial Calibration	WSC-CAM-V A, Table V A-1
Continuing Calibration	WSC-CAM-V A, Table V A-1
Method Blanks	WSC-CAM-V A, Table V A-1
Average Recovery	SW-846 Method 8000, Section 8.4
% Relative Standard Deviation	SW-846 Method 8000, Section 8.4
Surrogate Recovery	WSC-CAM-V A, Table V A-1
Internal Standards	WSC-CAM-V A, Table V A-1

Note: Because of the extensive analyte list and number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more analytes may not meet the performance standard for one or more QC elements. Under these circumstances, the analyst should attempt to locate and correct the problem and repeat the analysis for all nonconforming analytes. All nonconforming analytes along with the laboratory-specific acceptance criteria should be noted in the Initial Demonstration of Proficiency data provided.

It is essential that laboratory-specific performance criteria for LCS, LCS duplicate and surrogate recoveries also be calculated and documented as described in SW-846 Method 8000B, Section 8.7. When experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices, the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent quality control performance standards described in Table II A-1 to assess analytical trends (i.e., systematic bias, etc) and improve overall method performance by preempting potential non-conformances.

For SW-846 Method 8082, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table V A-1. It should be noted that the performance standards listed in Table V A-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory. Laboratories are encouraged to continually strive to minimize variability and improve the accuracy and precision of their analytical results. In some cases, the standard laboratory acceptance criteria for the various QC elements may require modification to accommodate more rigorous project-specific data quality objectives prescribed by the data user. The laboratory may be required to modify routine sample introduction and/or analytical conditions to accommodate project-specific data quality objectives.

This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GCs), and skilled in the interpretation of gas chromatograms for PCB analyses in environmental matracies. Each analyst must demonstrate the ability to produce acceptable quantitative and qualitative results for PCBs, reported as Aroclors or as individual PCB congeners with this method.

1.1.3 Sample Extraction/Cleanup Methods for SW-846 Method 8082

Sample Extraction and Concentration

Samples for analysis by SW-846 Method 8082 must be extracted or diluted using one of the following methods.

SW-846 Method	Matrix	Description
3542	Air Samples	Extraction of Analytes Collected Using a Modified Method 5 Sampling Train
3510C	Aqueous	Separatory Funnel liquid-Liquid Extraction
3520C	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3540C	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545A	Soil/Sediment	Pressurized Fluid Extraction (PFE)
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction (MSE)
3550C	Contaminated Solids[1]	Ultrasonic Extraction
3580A	NAPL	Solvent Dilution

1. Sonication may only be used for the extraction of highly contaminated (free product) non-soil/sediments (debris). Any other use of ultrasonic extraction is not allowed

Extract Cleanup

Extracts may be cleaned up by any of the following methods prior to GC analysis by SW-846 Method 8082. The recommended cleanup methods for routine PCB analyses are SW-846 Methods 3660B and 3665A.

SW-846 Method	Cleanup Method	Cleanup Type
3600 C	General Cleanup Selection	Not Applicable
3610 B	Alumina	Adsorption
3620 B	Florisil	Adsorption
3630 C	Silica Gel	Adsorption
3640 A	Gel-Permeation Cleanup (GPC)	Size-Separation
3660 B	Sulfur Cleanup	Oxidation/Reduction

1.2 Summary of Method

A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate matrix-specific sample extraction technique. Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), Method 3520C (continuous liquid-liquid extractor), or other appropriate technique. Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 C (Soxhlet), Method 3541 (automated Soxhlet), or other appropriate technique.

Extracts for PCB analysis should routinely be subjected to a sulfuric acid/potassium permanganate and sulfur cleanup using SW-846 Methods 3665A and 3660B, respectively. These cleanup techniques will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, SW-846 Method 8082 is not applicable to the analysis of these single component organochlorine or organophosphorus pesticides compounds and SW-846 Method 8081A is the method of choice for these pesticide compounds.

After cleanup, the extract is analyzed by injecting a 2-µL aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column equipped with either an electron capture or electrolytic conductivity detector.

The chromatographic data produced may then be used to identify and quantify the nine (9) Aroclors listed in Table V A-2, individual PCB congeners, or to determine total PCBs as the cumulative sum of individual Aroclors or congeners.

Identification of PCBs based on a single-column analysis should be confirmed on a second column, or should be supported by at least one other independent qualitative technique. Although a dual-column option may satisfy this requirement, due caution should be exercised when highly contaminated samples are processed or during times of high sample throughput.

Dual column confirmation is not required for samples with concentrations of all Aroclors and/or congeners below their respective reporting limit.

1.3 Interferences

Refer to SW-846 Methods 3500 (Sec. 3.0, in particular), 3600 C, and 8000 B for a detailed discussion of interferences.

Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories.

- Contaminated solvents, reagents, or sample processing hardware,
- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces,
- Non-target compounds simultaneously extracted from the sample matrix which cause a detector response, and
- Co-elution of target analytes

An in depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

1.3.1 Chemical Contaminants

Major contaminant sources for SW-846 Method 8082 include, but are not limited to, contaminated solvents and inadvertent contact of extraction fluids with rubber and/or plastic materials. The use of non-polytetrafluoroethylene (PTFE) thread sealants or plastic tubing should be avoided. <u>It should be noted that interfering contaminants may also be concentrated during sample preparation and cleanup</u>. Analyses of calibration and reagent blanks provide information about the presence of cross- contamination. When potential interfering peaks are noted in blanks, the analyst should review sample pretreatment and concentration procedures to evaluate the source of contamination.

Raw chromatographic data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem. **Subtracting blank values from sample results is not permitted.** If the laboratory determines that the concentration reported in the blank is so high that false positive results are likely in the associated samples, then the laboratory should fully explain this situation in the case narrative.

Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations by SW-846 Method 8082. Common flexible plastics contain varying amounts of phthalate esters, as plasticizers, which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents,

reagents and glassware may be required to eliminate background phthalate ester contamination. These materials can be removed through the use of SW-846 Cleanup Method 3665 A (sulfuric acid/permanganate cleanup).

[4]

1.3.2 Cross-Contamination

Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of PCBs. After the analysis of a sample containing high concentrations of PCBs, one or more blanks should be analyzed to check for potential cross-contamination/carryover. The concentration of PCBs which can cause cross-contamination/carryover must be determined by the laboratory and will be dependent upon the concentration and level of saturation of the particular analyte. Concentrations of PCBs which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample run. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections.

1.3.3 Elemental Sulfur Contamination

Elemental sulfur (S) is readily extracted from soil/sediment samples and may cause chromatographic interferences in the determination of PCBs by SW-846 Method 8082. Sulfur can be removed through the use of SW-846 Cleanup Method 3660B.

1.3.4 Special Precautions

Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized at laboratory drying oven temperatures and spread to other glassware. Due caution should be exercised when drying glassware used for the analysis of samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

1.4 QA/QC Requirements for SW-846 Method 8082

1.4.1 General Quality and Control Requirements for Determinative Chromatographic Methods

Refer to SW-846 Method 8000B for general procedures for all chromatographic methods, including SW-846 Method 8082. These requirements include that each laboratory should maintain a formal quality assurance program and records to document the quality of all chromatographic data.

Quality control procedures necessary to evaluate GC system operation may be found in Method 8000B, Sec. 7.0, and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. Instrument quality control and

method performance requirements for the GC/ECD system may be found in SW-846 Method 8082, Section 8.0 and 9.0, respectively.

1.4.2 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8082

Specific QA/QC requirements and performance standards for SW-846 Method 8082 are presented in Table V A-1. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying analytical and reporting requirements will provide an LSP with "Presumptive Certainty" regarding the usability of analytical data to support MCP decisions. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty", parties must:

(a) Comply with the procedures described and referenced in WSC-CAM–V A;

(b) Comply with the applicable QC analytical requirements prescribed in Table V A-1 for this test procedure;

(c) Evaluate, and narrate, as necessary, compliance with performance standards prescribed in Table V A-1 for this test method; and

(d) Adopt the reporting formats and elements specified in the CAM

In achieving the status of Presumptive Certainty, parties will be assured that analytical data sets:

- ✓ Satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data;
- ✓ May be used in a data usability assessment, and, if in compliance with all MCP Analytical Method standards, laboratory QC requirements, and field QC recommended limits and action levels, the data set will be considered usable data to support site characterization decisions made pursuant to the MCP; and
- ✓ May be used to support a data representativeness assessment.

Widespread adherence to the "Presumptive Certainty" approach will promote inter-laboratory consistency and provide the regulated community with a greater degree of certainty regarding the quality of data used for MCP decision-making. The issuance of these requirements and standards is in no way intended to preempt the exercise of professional judgement by the LSP in the selection of alternative analytical methods. However, parties who elect not to utilize the "Presumptive Certainty" option have an

obligation, pursuant to 310 CMR 40.0017 and 40.0191(2)(c), to demonstrate and document an overall level of (laboratory and field) QA/QC, data usability, and data representativeness that is adequate for and consistent with the intended use of the data.

1.4.3 Special Considerations for Multi-component Arochlor Mixtures

The identification of multi-component mixtures (i.e., Arochlors) is not based on a single peak, but rather on the characteristic peaks that comprise the "fingerprint" of the mixture, using both the retention times and shapes of the indicator peaks. If specific Arochlors are contaminants of concern at a disposal site, it is the responsibility of the LSP to request that these specific multi-component analyte spikes be included in the LCSs and MS/MSDs. All Arochlors are generally not included in LCSs or MS/MSDs.

Table V A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8082

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Retention Time Windows	Laboratory Analytical Accuracy	(1) Established prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method. (Section 7.6 of SW-846 8000)	No	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards. (2) Low standard must be ≤ reporting limit. (3) %RSD should be ≤20 or "r" should be ≥0.99. (4) *Aroclors:* 5-point calibration with 1016/1260 required; 5-point calibration for other Aroclors may be warranted based on site-specific conditions. *Congeners:* 5-point calibration must include all target congeners. (5) If regression analysis is used, the curve must not be forced through the origin. (6) *Aroclors:* For Aroclors which are not calibrated with 5-points, laboratory must perform single analysis of these Aroclors at the mid-point (50%) of the calibration curve. The Aroclor should be verified with a one point standard within 12 hrs when detected in a sample. (7) Curves must be verified by an independent ICV before analysis.	No	Recalibrate as required by method.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in case narrative. If the average response factor or linear regression is not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the case narrative with a list of the affected analytes
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Every 12 hours prior to samples or every 20 samples, whichever is more frequent, and at the end of the analytical sequence. (2) Concentration level near midpoint of curve. (3) *Aroclors:* CCAL with 1016/1260 required; CCAL for other Aroclors may be warranted based on site-specific conditions. *Congeners:* CCAL must include all target congeners. (4) Percent difference or percent drift should be ≤15.	No	(1) Perform instrument maintenance, reanalyze CCAL and/or recalibrate as required by method. (2) Reanalyze "associated samples" if beginning or closing CCAL exhibited low response and Aroclors were or were not detected in samples. (3) Reanalyze "associated samples" if beginning or closing CCAL exhibited high response and Aroclors were detected in the samples. NOTE: "Associated Samples" refers to all samples analyzed since the last acceptable CCAL.	Report exceedances in case narrative.

Table V A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8082

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be less than or equal to reporting limit.	Yes	Locate source of contamination; correct problem; re-extract associated samples if contaminants are present in the method blank.	(1) Report non-conformances in case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention (such as the case narrative), should qualify the sample results. (3) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level should be between low and mid-level standard. (4) *Aroclors:* LCS with 1016/1260 required; Additional LCS spiked with other Aroclors may be warranted based on site-specific conditions. *Congeners:* LCS must include all target congeners. (5) Matrix-specific (e.g., soil, water). (6) Percent recoveries must be between 40 -140. (7) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	Recalculate the percent recoveries. Check MS/MSD; if recoveries are acceptable in MS/MSD, nonconformance may be isolated to LCS. If recoveries are outside criteria in MS/MSD, re-extract associated samples.	(1) Report non-conformances in case narrative. (2) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (3) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Method Precision	(1) Analyzed with every batch or every 20 samples, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all Aroclors and congeners included in LCS (4) Recommended to be run immediately after LCS in analytical sequence. (5) Laboratory–determined percent recoveries must be between 40 – 140. (6) Matrix-specific (e.g., soil, water,. etc.) (7) Laboratory–determined Relative Percent Difference (RPD) must be ≤20 for waters and ≤30 for solids.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Narrate non-conformances

Table V A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8082

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Project Specific MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Extracted with every 20 samples at discretion of laboratory or if requested by data user. (2) Matrix-specific. (3) Prepared using standard source different than used for initial calibration. (4) Concentration level should be between low and mid-level standard. (5) *Aroclors:* MS/MSD with 1016/1260 required; Additional MS/MSD spiked with other Aroclors may be warranted based on site-specific conditions. *Congeners:* MS/MSD must include all target congeners. (6) Percent recoveries should be between 40-140. (7) RPDs should be ≤50 for Aroclors and ≤30 for congeners.	Yes (when requested by data user)	Check LCS; if recoveries acceptable in LCS, evaluate alternate cleanup techniques for samples associated with MS/MSD and/or narrate non-conformance.	.Report exceedances in case narrative.
Surrogates	Accuracy in Sample Matrix	(1) Minimum of 2, one that elutes at beginning of GC run and one that elutes at end of GC run. Recommended surrogates: *Aroclor analysis:* TCMX and DCB *Congener analysis:* TCMX or DBOFB and BZ198 (2) Percent recoveries must be between 30-150 for both surrogates. (3) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	If the same surrogate is outside limits on both columns, re-extract the sample. If both surrogates are outside limits on one column only, reanalyze the sample. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.	(1) Note exceedances in case narrative. (2) If re-extraction or reanalysis yields similar surrogate non-conformances, the laboratory should report results of both extractions or analyses. (3) If re-extraction or reanalysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction or reanalysis only. (4) If re-extraction or reanalysis is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial and re-extraction or reanalysis. (5) If sample is not re-extracted or reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.

Table V A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8082

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards *(Congeners only)*	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 1. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.	No	If internal standard is outside limits, reanalyze sample unless obvious interference present.	(1) Note exceedances in case narrative. (2) If reanalysis yields similar internal standard nonconformance, the laboratory should report both results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recovery, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of holding time and yields acceptable internal standard recovery, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	Inter-laboratory consistency	(1) *Aroclors:* Laboratory should quantitate all Aroclors with a minimum of three peaks. All peaks must be ≥25% of height of largest Aroclors peak. At least one peak must be unique to the Aroclor. (2) *Aroclors:* Laboratory should use the average calibration factor for each of three to five peaks from each concentration level to quantitate Aroclors 1016 and 1260. Laboratory should use the average calibration factor for each of three to five peaks from single point standard to quantitate remaining Aroclors (when only single-point standard analyzed). If 5-point calibration performed for other Aroclors, follow procedure for 1016 and 1260. Calculate concentration of Aroclor using each individual peak and calculate the average concentration of the three results to obtain the final Aroclor concentration. *Congeners:* Laboratory should use the average response factor of each congener. (3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive PCB results. The laboratory must report the higher of the two results unless obvious interference is present on one of the columns. In this case, the laboratory can report the lower result. All required QA/QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well.	No	NA	(1) If the RPD between the dual column results exceeds 40, the laboratory should qualify the sample results and/or note the exceedance in the case narrative. (2) If the average response factor or linear regression is not used for analyte quantitation (e.g. quadratic equation), this must be noted in the case narrative with a list of the affected analytes. NOTE: If the high RPD can be definitively attributed to interference on one of the two columns, the laboratory should report the lower value and provide a discussion in the case narrative that this approach was used.

Table V A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8082

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting Issues	NA	(1) The laboratory must report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory must report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. **NOTE: Laboratories shall not perform dilutions on samples due to sulfur interference. Laboratories must use a cleanup technique to reduce the presence of sulfur interference.**	Yes	NA	(1) Qualification of the data are required if reporting values below the sample-specific reporting limit.

GC = Gas Chromatography
MS/MSDs = Matrix Spikes/Matrix Spike Duplicates
%RSD = Percent Relative Standard Deviation

DCB = Decachlorobiphenyl
ICV = Initial Calibration Verification

"r" = Correlation Coefficient
RPDs = Relative Percent Differences
TCMX = Tetrachloro-m-xylene

DBOFB = Dibromooctafluorobiphenyl

1.5 MCP Analyte List for SW-846 Method 8082

The MCP analyte list for SW-846 Method 8082, presented in Table V A-2, is intended to be protective of human health and the environment. The list is comprised of seven (7) Aroclor mixtures, specifically identified in Section 1 of SW-846 Method 8082, and two (2) non-target Aroclor mixtures. Aroclors are multi-component mixtures of chlorine-substituted biphenyls. The two non-target mixtures, Aroclor 1262 and 1268, although not routinely included in the calibration of the method, may be encountered as contaminants at some sites. The GC/ECD chromatograms for these Aroclor mixtures are presented in Appendix V A-2. If encountered, the concentration of these Aroclor mixtures must be determined by comparison to a mid-range calibration standard.

This method also provides procedures for the determination of a subset of the possible 209 PCB congeners. Nineteen (19) of 209 possible PCB congeners, listed in Section 1 of SW-846 Method 8082, have been tested by this method. These congeners were chosen for testing by EPA because many of them are present in the most common Aroclor formulations, and ***not because of their toxicological significance***. Most, ***but not all***, of the remaining 209 potential PCB congeners can be identified/resolved and quantified using the GC columns and chromatographic conditions described in this method after an initial demonstration of proficiency. Congeners are mentioned in this guidance for informational purposes only and ***need not be evaluated*** in support of routine MCP decision making.

PCBs are regulated under the MCP either as specific Aroclor mixtures (Aroclors 1016, 1221, 1232, 1242, 1248, 1254 and 1260) or as PCB-N.O.S. (not otherwise specified, CAS Number 01336-36-3). The latter category includes ***all*** 1,1' Biphenyl chlorinated derivatives. The cumulative sum of all such congeners would be regulated under this CAS Number, PCB-N.O.S, as Total PCBs. At the discretion of the LSP requesting the analysis, the use of PCB congeners rather than Aroclors may be an appropriate analytical alternative under the following circumstances:

- Samples containing multiple Aroclors,
- Samples containing Aroclors that have been weathered by long exposure in the environment,
- Process samples containing Aroclors that have been subjected to degradation by destructive treatment technologies,
- Evaluations requiring greater accuracy and specificity at sites with known PCB contamination,
- Samples collected in support of comprehensive ecological risk assessments, and/or
- To provide more specific and accurate Total PCB contaminant concentrations in support of MCP Method 3 risk assessment evaluations.

The appropriate list of congeners to be evaluated should be determined by the LSP in consultation with the laboratory and other end users of the data (risk assessors, etc.) on a site-specific basis. Alternatively, "EPA Method 680, Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry (November 1985), Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory (EMSL), Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 04268" should be considered an option to resolve the aforementioned analytical complications. EPA Method 680 utilizes GC/MS operated in the Selected Ion Monitoring (SIM) mode to identify and quantify the various PCB homologues (same number of substituted chlorines). A summation of the individual PCB homologues may then be used to reliably determine Total PCBs.

1.6 Additional Reporting Requirements for SW-846 Method 8082

While it is not necessary to request and report all the SW-846 Method 8082 analytes listed in Table V A-2 to obtain Presumptive Certainty, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials. These assessment activities may include but are not limited to:

- ✓ Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- ✓ Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- ✓ Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- ✓ Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830

In a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Uncharacterized sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or

- ✓ Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

Note that a desire to avoid detection and quantitation of a contaminant that is present or likely present at a site above background levels is <u>not</u> a valid reason to limit an analyte list, and that such an action could constitute a criminal violation of MGL c. 21E.

In cases where a truncated list of method analytes is selected, laboratories must still employ the method-specific quality control requirements and performance standards associated with the requested analytes list to obtain Presumptive Certainty status.

The Reporting Limit (based on the concentration of the lowest calibration standard) for each contaminant of concern must be less than or equal to the MCP standards or criteria that the

contaminant concentrations are being compared to (e.g., Method 1 Standards, RfDs, benchmark values, background, etc.). Meeting "MCP program" reporting limits may require analytical modifications, such as increased sampling weight or volume to increase sensitivity. All such modifications must be described in the Environmental Laboratory case narrative.

Table V A-2 SW-846 Method 8082 Analyte List

Analyte	CASN	MCP CLEANUP STANDARD	
		GW-3	S-1/GW-3
		µg/L (ppb)	µg/g (ppm)
Aroclor Mixtures:			
Aroclor 1016	12674-11-2	0.3	2
Aroclor 1221	11104-28-2	0.3	2
Aroclor 1232	11141-16-5	0.3	2
Aroclor 1242	53469-21-9	0.3	2
Aroclor 1248	12672-29-6	0.3	2
Aroclor 1254	11097-69-1	0.3	2
Aroclor 1260	11096-82-5	0.3	2
Aroclor 1262 [1]	37324-23-5	0.3 [2]	2 [2]
Aroclor 1268 [1]	11100-14-4	0.3 [2]	2 [2]

1. Non-target Aroclor mixture. Not usually included on MCP Analyte List for SW-846 Method 8082.
2. Not specifically listed in Subpart P, Massachusetts Oil and Hazardous Material list (MOHML) but regulated as PCB-N.O.S. (not otherwise specified, CAS Number 01336-36-3)

2.0 Data Usability Assessment for SW-846 Method 8082

Overall data usability is influenced by uncertainties associated with both sampling and analytical activities. This document provides detailed quality control requirements and performance standards for SW-846 Method 8082 which may be used to assess the analytical component of data usability. The sampling component of data usability, an independent assessment of the effectiveness of sampling activities to meet data quality objectives, is not substantively addressed in this document.

3.0 Reporting Requirements for SW-846 Method 8082

3.1 General Reporting Requirements for SW-846 Method 8082

General environmental laboratory reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VII A, Section 2.4. This guidance document provides recommendations for field QC, as well as the required content of the Environmental Laboratory Report, including:

- Laboratory identification information presented in WSC-CAM-VII A, Section 2.4.1,
- Analytical results and supporting information in WSC-CAM-VII A, Section 2.4.2,
- Sample- and batch-specific QC information in WSC-CAM-VII A, Section 2.4.3,
- Laboratory Report Certification Statement in WSC-CAM-VII A, Section 2.4.4,
- Copy of the Analytical Report Certification Form in WSC-CAM-VII A, Exhibit VII A-1,
- Environmental Laboratory Case Narrative contents in WSC-CAM-VII A, Section 2.4.5,
- Chain of Custody Form requirements in WSC-CAM-VII A, Section 2.4.6

3.2 Specific Reporting Requirements for SW-846 Method 8082

Specific Quality Control Requirements and Performance Standards for SW-846 Method 8082 are presented in Table V A-1. Specific reporting requirements for SW-846 Method 8082 are summarized below in Table V A-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements should always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data are to be available for review during an audit and may also be requested on a client-specific basis

Table V A-3 Routine Reporting Requirements for SW-846 Method 8082

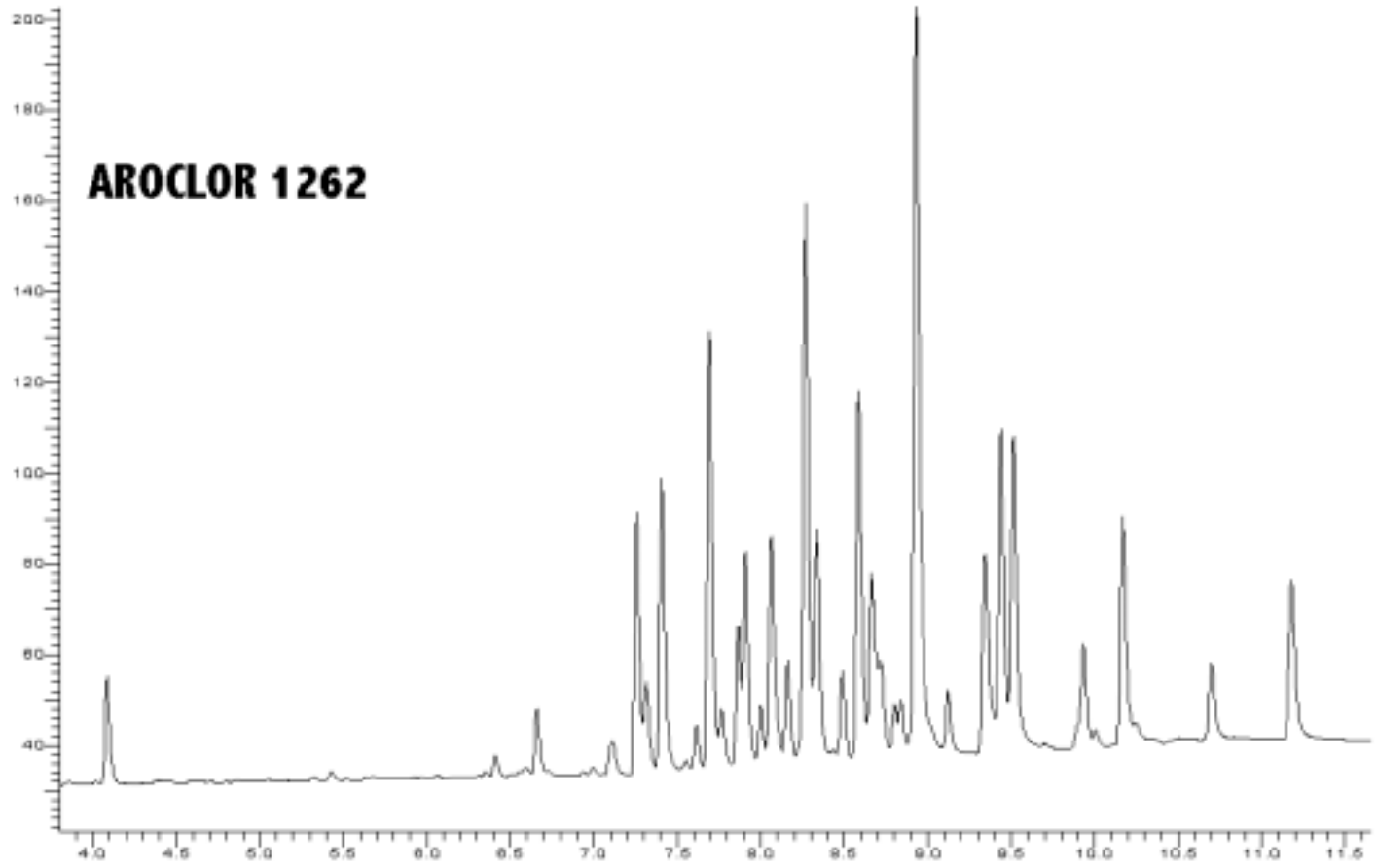
Parameter	Required Analytical Deliverable
Retention Time Windows	***NO***
Initial Calibration	***NO***
Continuing Calibration (CCAL)	***NO***
Method (Preparation) Blank	***YES***
Laboratory Control Spikes (LCSs)	***YES***
LCS Duplicate	***YES***
Matrix Spike (MS)	***YES (if requested by data user)***
Matrix Spike Duplicate (MSD)	***YES (if requested by data user)***
Matrix Duplicate (MD)	***YES (if requested by data user)***
Surrogates	***YES***
Internal Standards (ISs)	***NO***
Identification and Quantification	***NO***
General Reporting Issues	***YES***

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for PCBs analyzed by SW-846 Method 8082 in support of MCP decision-making are summarized below and presented in Appendix VII-A of WSC-CAM-VIIA, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)".

{PRIVATE } Matrix	Container [1]	Preservation [2]	Holding Time [3]
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to 4°C	7 days to extraction; 40 days from extraction to analysis
Aqueous Samples, with Residual Chlorine [4]	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) [5]. Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to 4°C	7 days to extraction; 40 days from extraction to analysis
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap	Cool to 4°C	14 days to extraction; 40 days from extraction to analysis
Waste Samples	(1) 500 mL amber wide mouth jar with a teflon lined screw cap.	No special preservation required	14 days to extraction; 40 days from extraction to analysis

1. The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis
2. Alternatively, soil samples for PCB analyses may be held for up to one (1) year if frozen within 24 hours of collection at < -10°C. Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing. Preparation or extraction must be commenced within 24 hours of thawing. Temperature must never be allowed to go below – 20 °C to avoid damage to seals, etc.
3. Holding time begins from time of sample collection.
4. Presence of residual chlorine is usually associated with drinking water samples
5. Confirm dechlorination. If Residual Chlorine > 5 mg/L additional dechlorination agent may be required

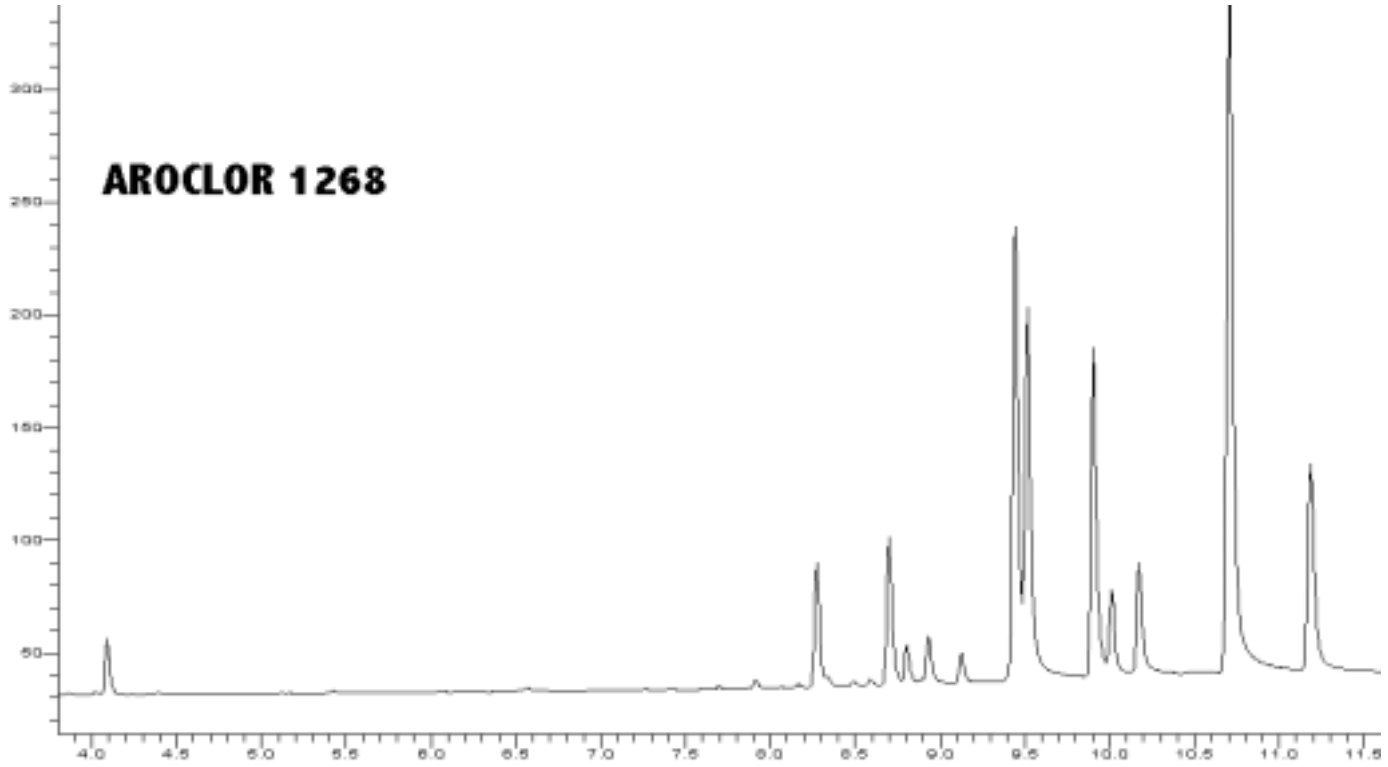
Exhibit V A-1 GC/ECD Chromatogram[1] for Aroclor 1262



System Parameters	Chromatographic Conditions
Injection: Splitless	Initial Temperature: 120 C^o
Injection Volume: 2.0 uL	Initial Hold: 0.00 Minutes
Detector: ECD	Ramp 1: 25 C^o/min to 220 C^o
Injector Temperature: 220 C^o	Ramp 2: 13 C^o/min to 300 C^o
Detector Temperature: 370 C^o	Final Hold: 2.15 min
Carrier Gas: Hydrogen	
Carrier Gas Flow: I mL/min	**Total Run Time: 12.3 min**

1. Chromatogram for Aroclor 1262 provided by Woods Hole Group Environmental Laboratories, 375 Paramount Drive, Suite 2, Raynham, MA 02767

Exhibit V A-2 GC/ECD Chromatogram[1] of Aroclor 1268



System Parameters	Chromatographic Conditions
Injection: Splitless	Initial Temperature: 120 C^o
Injection Volume: 2.0 uL	Initial Hold: 0.00 Minutes
Detector: ECD	Ramp 1: 25 C^o/min to 220 C^o
Injector Temperature: 220 C^o	Ramp 2: 13 C^o/min to 300 C^o
Detector Temperature: 370 C^o	Final Hold: 2.15 min
Carrier Gas: Hydrogen	
Carrier Gas Flow: I mL/min	**Total Run Time: 12.3 min**

1. Chromatogram for Aroclor 1268 provided by Woods Hole Group Environmental Laboratories, 375 Paramount Drive, Suite 2, Raynham, MA 02767